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Final Project Report of the Determination of Alpha Activity in Mud

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Carbide and Carbon Chemicals Corporation, Operating Contractor for the U.S. Atomic Energy Commission.

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Final Project Report of the

Determination of Alpha Activity in Mud

ABSTRACT

It was requested of the Counting Section by the Health Physics Department to determine in mud, the alpha activity due to uranium. A method using di-ethyl ether was developed for extracting the uranium from the mud for alpha counting. This method gives reasonable accuracy and precision for concentrations as low as one part of uranium oxide per million parts of mud.

Final Project Report of the

Determination of Alpha Activity in Mud

It was requested of the Counting Section by the Health Physics Department to determine in mud the alpha activity due to uranium. Since the mud itself was an extremely high alpha absorber and the expected activity was small, it was obvious that a separation of the uranium from the mud would be essential for precise counting.

A method for extraction and purification of uranium with di-ethyl ether had been used for some time in the Counting Section, and it was decided to try to apply this method to the recovery of uranium in the concentrations of one part of uranium oxide per one million parts of mud. To detect with accuracy uranium in this low concentration in the development work, it was decided to employ, as a tracer, extremely alpha active uranium enriched in the 234 and 235 isotopes emitting 140 alpha counts/min. per microgram/$\frac{1}{2}$ solid angle. The activity of normal uranium is only 0.64 cts/min/microgram. For the development work samples of ten micrograms of uranium oxide tracer in solution was added to ten grams of mud, thoroughly mixed, and dried. The mud for this work was obtained from the banks of the Clinch River above Oak Ridge, since it should contain no active materials from the Oak Ridge area. The mud was dried, ground, and passed through a 100 mesh sieve. The number of counts from the uranium recovered from the mud were compared to the number of counts introduced by the tracer.

EXPERIMENTAL

The organic material in the mud is first removed by oxidation with nitric and perchloric acids. It was found that sulfuric acid with nitric was entirely

unsuitable since it caused an undesirable precipitate at a latter step in the purification. After the organic material is destroyed, the acid insoluble material is removed by centrifuging and the solution is evaporated and fumed until all the perchloric acid is removed. The residue is brought into solution with a minimum amount of hydrochloric acid and repeatedly evaporated with nitric acid to remove the chlorides. To the remaining nitrate solution, saturated acid ammonium nitrate solution, a small amount of concentrated nitric acid, and one gram of tartaric acid are added. The tartaric acid complexes the iron preventing its extraction by the di-ethyl ether as ferric chloride. The uranium in the resulting solution is extracted with di-ethyl ether in separatory funnels. The uranyl nitrate is re-extracted from the di-ethyl ether with water. The water solution is evaporated to dryness. The residue is taken up in a few drops of nitric acid and saturated acid ammonium nitrate solution and re-extracted as before. This is necessary to remove the last traces of iron that extracts with the uranium. If, after the first extraction, no visible evidences of iron is present in the dry beaker the second extraction can be omitted. After the second extraction the evaporated water solution is taken up with nitric acid and evaporated to dryness until all organic matter is oxidized. The residue is taken up in one drop of concentrated nitric followed by 5 ml. of distilled water and transferred to a plating cell where it is plated according to the routine procedure and alpha counted.

DATA AND RESULTS

Below is listed the number of counts from the uranium recovered from 10 grams of mud to which ten milliliters of tracer solution, containing ten micrograms of U_3O_8, had been added. Also listed below is the counts from 10 milliliters of tracer solution transferred directly to a plating

The following table gives the results of nineteen determinations:

Counts from the Uranium Tracer recovered from Mud		Counts from direct plating of Uranium Tracer
1136	1421	1418
1318	1327	1422
1302	1355	1405
1236	1265	1408
1135	1080	$\bar{x}$ = 1413
1416	1235	
1282	1100	
1209	1261	
1225	1196	
1429	$\bar{x}$ = 1259	

The value of the tracer was found to be 1413 $\pm$ 13 alpha counts a minute. The average value of the tracer recovered from the mud was found to be 1259 $\pm$ 50 alpha counts per minute. 89.1% $\pm$ 3.6% of the tracer was the average recovery from the mud. On a single determination, the recovery is 89% with a precision of $\pm$ 17%.

DISCUSSION

Some work was done using penta-ether as an extraction agent but it was found that it was not superior to the di-ethyl ether, was much slower to separate from the aqueous solution, and formed undesirable emultions more easily. The dissolved penta-ether could not be evaporated from the water extraction prior to plating and its removal could be effected only by oxidation with nitric or perchloric acids. The di-ethyl ether gave much cleaner extractions.

To get the recovery desired it was found that the nitrate ion concentration should be as high as possible for quantitative extraction. Also great care should be exercised to prevent iron in the mother solution from being mechanically carried through the extraction in emultions and on the walls of the vessel. A small amount of nitric acid was found effective in breaking emultions.

This procedure can also be applied to the determination of alpha activity due to uranium in sewage disposal plant sludge if special precautions are taken in the initial removal of organic material. Since the sludge has a much higher organic content than mud, perchloric acid can not be used as an explosion may result. Most of the organic matter is first destroyed by nitric acid after which perchloric can be safely added. From thereon the procedure is the same as for mud.

It was found that electroplating the uranium extracted from the mud gave approximately twenty percent better recovery than the residue method and for this reason plating has been adopted in the procedure.

CONCLUSION

A method has been developed whereby it is possible to determine the alpha activity, due to uranium, in mud at concentrations as low as one part per million with a reasonable degree of precision and accuracy. It is felt possible that by this method a concentration as low as 170 parts of normal uranium per one billion parts of mud could be detected.

ACKNOWLEDGEMENTS

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